Organometallics **2000,** *19,* 2389−2392

Notes

Synthesis, Structure, and Fluxionality of (μ-Diphenylsilylene)(fulvalene)tetracarbonyldirhenium— Double Si−H Activation to the First Fulvalene Silylene Complex

Bernd Kayser, Michael J. Eichberg, and K. Peter C. Vollhardt*

Department of Chemistry, University of California at Berkeley, and the Chemical Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720-1460

Received January 10, 2000

*Summary: (Fulvalene)hexacarbonyldirhenium (**1**) furnishes the first silylene fulvalenedimetal complex **3** by double Si−H activation. The solid-state structure of the molecule was determined by X-ray analysis and its solution dynamics by ^{1}H NMR spectroscopy.*

Introduction

Silylene complexes of transition metals have received considerable attention because of their (potential) intermediacy in a range of metal-catalyzed organosilicon transformations.[1] Their dinuclear variants[2] are of interest as models on which to search for potential cooperative effects that might be relevant to heterogeneous catalysis. We are investigating the fulvalene (Fv) ligand as a matrix used to anchor two metals in close proximity in order to probe for novel reactivity.[3] As part of an effort aimed at effecting multiple C−H activations on FvM_2 systems, we turned to $[FvRe_2(CO)_6]$ (**1**) and report here a model study featuring double Si−H activation to give the title compound. Complex **1** has been made previously[4] and remains virtually unexplored,[5] which is surprising, considering the rich chemistry of $[CpRe(CO)_3]$.[6] Of particular relevance to the present study is the photolysis of the latter in the presence of silanes which results in decarbonylated products of oxidative addition, $[CpRe(CO)_2(SiR_3)H]$.[7]

Results and Discussion

Irradiation of **1** in benzene containing 3 equiv[7a] of diphenylsilane for 15 h led to the complete disappearance of starting materials (Scheme 1). A thin film of decomposition products was evident on the surface of the glass vessel, but the solution remained clear. Monitoring the progress of this spectroscopically clean transformation by ^{1}H NMR spectroscopy revealed the appearance of signals ascribable to **3** and an (unisolated) intermediate assigned structure **2**, by comparison with spectral data obtained for similar CpRe compounds.[2,7,8] Most indicative are the hydride signals at δ −11.61 (Re−H) and 6.60 ppm (Si−H), as well as the expected eight Fv signals (δ 2.61, 3.04, 3.57, 4.00, 4.22, 4.31, 4.61, and 4.66 ppm). Continued irradiation transformed **2** into **3**, purified by column chromatography on silica. Silylene complex **3** is an air-stable, yellow, crystalline solid that is only poorly soluble in organic solvents, such as C_6H_6, CH_3CN, acetone, CH_2Cl_2, $CHCl_3$, and diethyl ether, preventing the measurement of ^{13}C and ^{29}Si NMR spectra. Its structure is in accord with the available spectral data. Thus, the mass spectrum exhibited an M^+ peak at m/z 794 with the appropriate isotopic pattern and the IR spectrum the expected four terminal carbonyl bands. ^{1}H NMR spectroscopy revealed the presence of four broad (vide infra) Fv peaks, at δ 2.94, 3.76, 4.52, and 4.62 ppm, and two signals for the ortho phenyl hydrogens (δ 7.50, 7.83 ppm) all integrated for 2H each, in addition to a multiplet for the remaining aromatic hydrogens. Finally, the electronic spectrum featured two bands at 283 and 314 nm.

---

* To whom correspondence should be addressed. Telephone: 510-642-0286. Fax: 510-643-5208. E-mail: vollhard@cchem.berkeley.edu.

[1] (a) Corey, J. Y.; Braddock-Wilking, J. *Chem. Rev.* **1999**, *99*, 175. (b) Gauvin, F.; Harrod, J. F.; Woo, H. G. *Adv. Organomet. Chem.* **1998**, *42*, 363. (c) Pannell, K. H.; Sharma, H. K. *Chem. Rev.* **1995**, *95*, 1351. (d) Lickiss, P. D. *Chem. Soc. Rev.* **1992**, 271. (e) Zybill, C. *Top. Curr. Chem.* **1991**, *160*, 1. (f) Tilley, T. D. In *The Silicon-Heteroatom Bond*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1991; Chapters 9 and 10, pp 245, 309. (g) Tilley, T. D. *Comments Inorg. Chem.* **1990**, *10*, 37. (h) Tilley, T. D. In *The Chemistry of Organic Silicon Compounds*; Patai, S., Rappoport, Z., Eds.; Wiley: New York, 1989; Chapter 24, p 1415. (i) Curtis, M. D.; Epstein, P. S. *Adv. Organomet. Chem.* **1981**, *19*, 213.

[2] Ogino, H.; Tobita, H. *Adv. Organomet. Chem.* **1998**, *42*, 223.

[3] See, inter alia: (a) Vollhardt, K. P. C.; Cammack, J. K.; Matzger, A. J.; Bauer, A.; Capps, K. B.; Hoff, C. D. *Inorg. Chem.* **1999**, *38*, 2624. (b) Boese, R.; Cammack, J. K.; Matzger, A. J.; Pflug, K.; Tolman, W. B.; Vollhardt, K. P. C.; Weidman, T. W. *J. Am. Chem. Soc.* **1997**, *119*, 6757. (c) Tilset, M.; Vollhardt, K. P. C.; Boese, R. *Organometallics* **1994**, *13*, 3146. (d) McGovern, P. A.; Vollhardt, K. P. C. *Synlett* **1990**, 493.

[4] (a) Rausch, M. D.; Spink, W. C.; Conway, B. G.; Rogers, R. D.; Atwood, J. L. *J. Organomet. Chem.* **1990**, *383*, 227. (b) Nesmeyanov, A. N.; Kolobova, N. E.; Anisimov, K. N.; Makarov, Yu. V. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1969**, 357.

[5] Herberhold, M.; Biersack, M.; Bitterwolf, T. E.; Rheingold, A. L. *Z. Naturforsch.* **1993**, *48B*, 161.

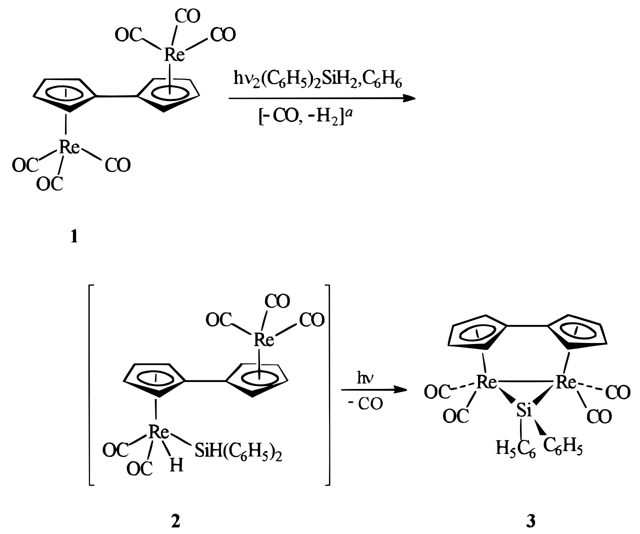
[6] Connor, J. M. In *Comprehensive Organometallic Chemistry II*; Abel, E. W., Stone, F. G. A., Wilkinson, G., Eds.; Pergamon: Oxford, U.K., 1995; Vol. 6, Chapter 9 (Low Valent Organorhenium Compounds), p 167.

[7] (a) Dong, D. F.; Hoyano, J. K.; Graham, W. A. G. *Can. J. Chem.* **1981**, *59*, 1455. (b) Hoyano, J. K.; Graham, W. A. G. *Organometallics* **1982**, *1*, 783.

[8] Sakaba, H.; Ishida, K.; Horino, H. *Chem. Lett.* **1998**, 149.

10.1021/om000022u CCC: $19.00

Scheme 1



[a] Not explicitly detected.

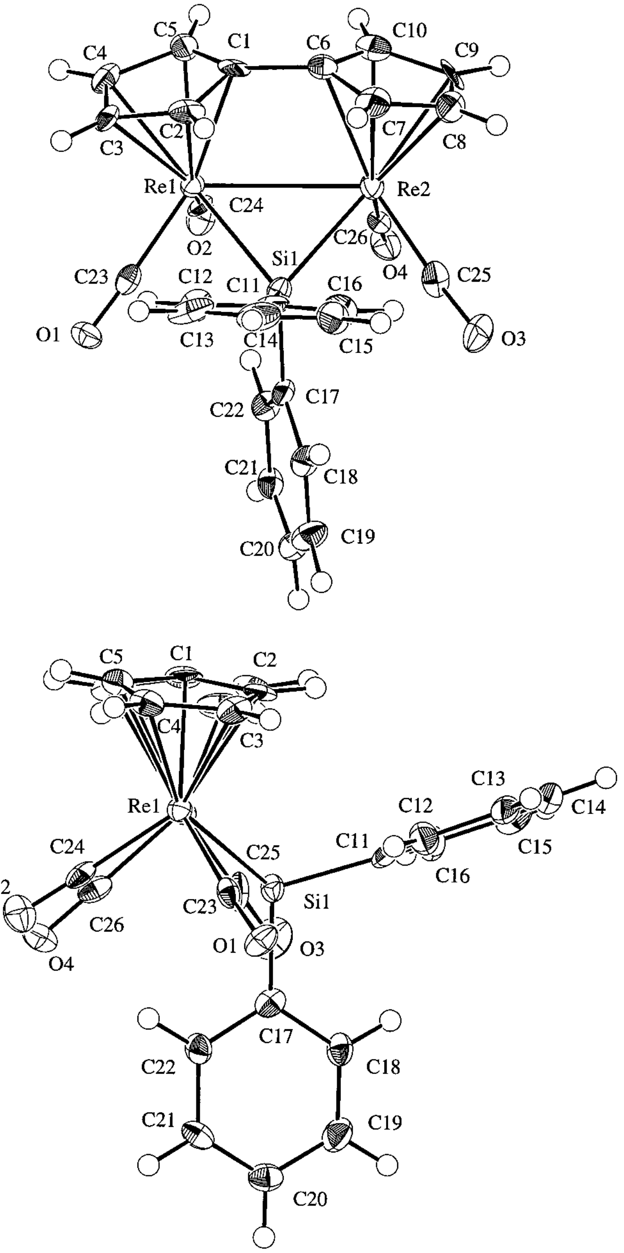


Figure 1. Molecular structure of **3**: views perpendicular (top) and parallel (bottom) to the Re–Re bond. Elements are represented by thermal ellipsoids at the 50% level. Selected bond lengths (Å) and angles (deg): Re(1)–Re(2) = 2.9926(4), Re(1)–Cp(centroid) = 1.9400(4), Re(2)–Cp(centroid) = 1.9619(5), Re(1)–Si(1) = 2.446(3), Re(2)–Si(1) = 2.455(3), Si(1)–C(11) = 1.90(1), Si(1)–C(17) = 1.862(10), C(1)–C(6) = 1.47(1); Re(2)–Re(1)–Si(1) = 52.51(6), Re(2)–Re(1)–Cp(centroid) = 103.19(1), Si(1)–Re(1)–Cp(centroid) = 126.07(6), Re(1)–Re(2)–Si(1) = 52.22(6), Re(1)–Re(2)–Cp(centroid) = 101.86(2), Si(1)–Re(2)–Cp(centroid) = 129.04(6), Re(1)–Si(1)–Re(2) = 75.27(8), Re(1)–Si(1)–C(11) = 115.3(3), Re(1)–Si(1)–C(17) = 120.9(3), Re(2)–Si(1)–C(11) = 116.7(3), Re(2)–Si(1)–C(17) = 119.1(3), C(11)–Si(1)–C(17) = 107.3(4).

Rigorous structural proof of **3** was provided by an X-ray analysis, using crystals obtained by slow cooling of a saturated solution in acetone (Figure 1 and Table 1). The result shows the connectivity depicted in **3**, in particular the diphenylsilylene fragment, which constitutes the apex of a dirhenasilacyclopropane ring. Significantly, the plane of the latter forms a dihedral angle with that defined by the roughly coplanar Re(1)–Re(2)–C(6)–C(1) of ca. 40° (torsion angles Si–Re(1)–C(1)–C(6) = 42.5 (7)° and Si–Re(2)–C(6)–C(1) = −37.2 (7)°); i.e., the silicon is located "to the side" of the $FvRe_2$ core, leading to the partial desymmetrization already indicated by the ^1H NMR and IR data. Most strikingly, however, the three-membered ring is almost coplanar with the proximal $Re_2(CO)_2$ unit (torsion angles Si–Re(2)–Re(1)–C(23) = −19.4 (4)°, Si–Re(1)–Re(2)–C(25) = 14.5(4)°), a structural feature that may be connected to the compound's fluxional behavior (vide infra). The silicon is positioned quite symmetrically, with respective angles and distances Re(2)–Re(1)–Si = 52.51(6)°, Re(1)–Re(2)–Si = 52.22(6)°, Re(1)–Si = 2.446(3) Å, and Re(2)–Si = 2.455 (3) Å, the last two bonds slightly shorter than that in $\{cis\text{-}CpRe(CO)_2(H)[Si(C_6H_5)_3]\}$ (2.49(1) Å).[9] The Re–Si–Re angle is 75.27(8)°, at the high end, but within the range, of other μ-SiR_2–dimetal compounds.[2] The Re–Re bond (2.9926 (4) Å) is slightly longer than those in $[Cp_2Re_2(CO)_5]$ (2.957(1) Å)[10] and $[Cp^*(CO)_2Re(\mu\text{-}CO)Re(CO)(CH_3CN)Cp^*]$ (2.951(2) Å)[11] but shorter than those in, for example, $[Cp^*_2Re_2(CO)_4(\mu\text{-}H)_2]$ (3.143(1) Å),[12] $[Cp^*_2Re_2(CO)_4(\mu\text{-}\eta^1{:}\eta^1\text{-}CH_3O_2CC{\equiv}CCO_2CH_3)]$ (3.0927(8) Å),[13] and $[Cp(\mu\text{-}\eta^1{:}\eta^5\text{-}C_5H_4)Re_2(CO)_4C_6F_4H]$ (3.0258(7) Å).[14] Similar to other metal–metal-bonded Fv systems,[3] the two Cp rings are twisted (3°) and bent (26.04°) from (co)planarity. Finally, the phenyl moieties attached to Si are almost at right angles (94.7°) to each other, presumably for steric reasons.

Bridged silylene (and related germylene) complexes exhibit a range of dynamic behavior, including fluxionality, in the NMR experiment.[2] These dynamics are associated with a variety of processes, such as hindered

---

[9] Smith, R. A.; Bennett, M. J. *Acta Crystallogr.* **1977**, *B33*, 1113.

[10] Foust, A. S.; Hoyano, J. K.; Graham, W. A. G. *J. Organomet. Chem.* **1971**, *32*, C65.

[11] Casey, C. P.; Cariño, R. S.; Sakaba, H.; Hayashi, R. K. *Organometallics* **1996**, *15*, 2640.

[12] Casey, C. P.; Sakaba, H.; Hazin, P. N.; Powell, D. R. *J. Am. Chem. Soc.* **1991**, *113*, 8165.

[13] Casey, C. P.; Cariño, R. S.; Hayashi, R. K.; Schladetzky, K. D. *J. Am. Chem. Soc.* **1996**, *118*, 1617.

[14] Godoy, F.; Higgitt, C. L.; Klahn, A. H.; Oelckers, B.; Parsons, S.; Perutz, R. N. *J. Chem. Soc., Dalton Trans.* **1999**, 2039.

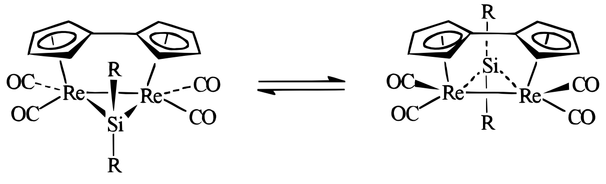
Table 1. Crystal and Data Collection Parameters for $FvRe_2(CO)_4[\mu\text{-}Si(C_6H_5)_2]$ (3)

formula	$C_{26}H_{18}SiO_4Re_2$
fw	794.93
cryst dimens, mm	0.20 × 0.15 × 0.09
cryst syst	orthorhombic
space group	*Pbca* (No. 61)
temp, °C	−130.0
a, Å	15.0034(5)
b, Å	17.1665(6)
c, Å	17.6279(6)
v, Å^3	4540.2(2)
D_c, g cm^{-3}	2.326
Z	8
radiation (λ, Å)	Mo Kα (0.710 69)
no. of total rflns	22 040
no. of unique rflns	4646
no. of obsd rflns	2074 ($I > 3.00\sigma(I)$)
no. of params	298
μ, cm^{-1}	107.44
R[a]	0.024
R_w[b]	0.028

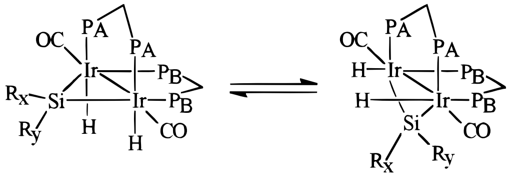
[a] $R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$. [b] $R_w = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

(aryl) substituent mobility,[15] reversible metal−SiH interactions,[16] phosphine dissociation−association,[17] and monomer−dimer equilibria.[18] Perhaps more relevant to the behavior of **3** (vide infra) are several reports of reversible inversion at Si[8,16a,19] and at either or both of the two attached metals[20] (notably cis−trans isomerization of Cp in Cp_2M_2 systems[19c,21]), for which a number of mechanisms have been postulated,[3] among them intermediate terminal silylene generation,[19b−d,20b] M−M bond cleavage,[21b] and converse Berry pseudorotation.[20b] To what extent these processes are coupled is still a matter of dispute,[3] and the dynamics of **3**, in which the two Cp ligands are connected, could shed some light on this problem.

Indeed, as indicated above, the ^1H NMR signals of **3** at room temperature are broadened, suggesting mobility on the NMR time scale, confirmed by a variable-temperature study. Thus, all signals sharpened at low temperature and coalescence of the four Fv (to two) as well as the two ortho phenyl signals (to one) was observed at high temperatures (307, 325, and 298 K, respectively). The coalescence temperature of 307 K of the two low-field Fv signals was used to estimate a $\Delta G^‡ = 15.2 \pm 0.5$ kcal mol^{-1}.[22] The clearly correlated coalescence of the signals of the two parts of the molecule can be explained by invoking either reversible bridging to terminal silylene/carbonyl migration in a "merry-go-round" fashion (as in $[Cp_2Re_2(CO)_5]$),[23] in which the terminal silylene undergoes well-anticipated rapid C−Si bond rotation,[3,24] or more simply (and, hence, perhaps more attractive)[25] a double-turnstile motion at the respective metals, equivalent to double inversion or a "swinging through" of the silylene group from one side of the Fv ligand to the other, as in **3a** ⇌ **3b**. This pathway has precedence in the similar fluxional behavior of $FvMo_2(CO)_4(RC{\equiv}CR)$ complexes ($\Delta G^‡ \approx 15$ kcal mol^{-1})[26] and the topologically equivalent rapid equilibration **4a** ⇌ **4b** in $[Ir_2(H)_2(CO)_2(\mu\text{-}SiR_2)(dppm)_2]$.[20a]



3a R = C_6H_5 **3b**



4a P = $P(C_6H_5)_2$ **4b**

Its relative facility may be indicated by the relevant geometric features of **3** (vide supra), suggesting a ground state that is "activated" with respect to the postulated movement. Finally, consistent with the absence of terminal silylene intermediates is the lack of reactivity of **3** with potential donor interceptors, such as amines, nitriles, and ketones.[1]

Experimental Section

General Information. All manipulations were carried out in dry solvents under argon. Commercially available diphenylsilane was used without further purification, and $[FvRe_2(CO)_6]$ was prepared according to or in adaptation of literature procedures.[5,27] The photoreaction was carried out with a broad-band Hanovia 450 W medium-pressure Hg-vapor lamp fitted with a water-cooled jacket. NMR: Bruker AM 400, with tetramethylsilane as internal standard. IR: Perkin-Elmer Model 2000 IR. Mass spectra were acquired by the UC Berkeley Mass Spectral Facility.

---

(15) (a) Malisch, W.; Hindahl, K.; Käb, H.; Reising, J.; Adam, W.; Prechtl, F. *Chem. Ber.* **1995**, *128*, 963. (b) Tobita, H.; Shinagawa, I.; Ohnuki, S.; Abe. M.; Izumi, H.; Ogino, H. *J. Organomet. Chem.* **1994**, *473*, 187.

(16) (a) Rosenberg, L.; Fryzuk, M. D.; Rettig, S. J. *Organometallics* **1999**, *18*, 958. (b) Fryzuk, M. D.; Rosenberg, L.; Rettig, S. J. *Organometallics* **1996,** *15*, 2871. (c) Wang, W.-D.; Eisenberg, R. *J. Am. Chem. Soc.* **1990**, *112*, 1833. (d) Suzuki, H.; Takao, T.; Tanaka, M.; Moro-oka, Y. *J. Chem. Soc., Chem. Commun.* **1992**, 476.

(17) Kim, Y.-J.; Lee, S.-C.; Park, J.-I.; Osakada, K.; Choi, J.-C.; Yamamoto, T. *Organometallics* **1998**, *17*, 4929.

(18) Heyn, R. H.; Tilley, T. D. *J. Am. Chem. Soc.* **1992**, *114*, 1917.

(19) See, inter alia: (a) Campion, B. K.; Heyn, R. H.; Tilley, T. D. *Organometallics* **1992**, *11*, 3918. (b) Tobita, H.; Kawano, Y.; Ogino, H. *Chem. Lett.* **1989**, 2155. For a particularly relevant Ge analogue, see: (c) Job, R. C.; Curtis, M. D. *Inorg. Chem.* **1973**, *12*, 2514.

(20) (a) McDonald, R.; Cowie, M. *Organometallics* **1990**, *9*, 2468. For a seminal study of a Ge analogue, see: (b) Adams, R. D.; Cotton, F. A. *J. Am. Chem. Soc.* **1970**, *92*, 5003.

(21) (a) Kawano, Y.; Tobita, H.; Shimoi, M.; Ogino, H. *J. Am. Chem. Soc.* **1994**, *116*, 8575. (b) Ueno, K.; Hamashima, N.; Ogino, H. *Organometallics* **1992**, *11*, 1435. (c) Kawano, Y.; Tobita, H.; Ogino, H. *Organometallics* **1992**, *11*, 499. (d) Ueno, K.; Hamashima, N.; Shimoi, M.; Ogino, H. *Organometallics* **1991**, *10*, 959. (e) Malisch, W.; Ries, W. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 120. For a relevant early study of a related Ge compound, see: (f) Adams, R. D.; Brice, M. D.; Cotton, F. A. *Inorg. Chem.* **1974**, *13*, 1080.

(22) Braun, S.; Kalinowski, H.-O.; Berger, S. *100 and More Basic NMR Experiments*; VCH: Weinheim, Germany, 1996; p 120.

(23) Lewis, L. N.; Caulton, K. G. *Inorg. Chem.* **1981**, *20*, 1139.

(24) For recent references, see: (a) Arnold, F. P. A., Jr. *Organometallics* **1999**, *18*, 4800. (b) Ueno, K.; Masuko, A.; Ogino, H. *Organometallics* **1999**, *18*, 2694. (c) Grumbine, S. K.; Mitchell, G. P.; Straus, D. A.; Tilley, T. D. *Organometallics* **1998**, *17*, 5607. (d) Wada, H.; Tobita, H.; Ogino, H. *Chem. Lett.* **1998**, 993.

(25) Hoffmann, R.; Minkin, V. I.; Carpenter, B. K. *Bull. Soc. Chim. Fr.* **1996**, *133*, 117.

(26) Drage, J. S.; Vollhardt, K. P. C. *Organometallics* **1986**, *5*, 280.

(27) Herrmann, W. A.; Andrejewski, D.; Herdtweck, E. *J. Organomet. Chem.* **1987**, *319*, 183.

Preparation of 3. An argon-flushed quartz Schlenk tube was charged with [FvRe$_2$(CO)$_6$] (200 mg, 0.30 mmol). Dry benzene (30 mL) and diphenylsilane (168 μl, 0.90 mmol) were added; the resulting solution was degassed with argon for 5 min and then irradiated for 24 h at room temperature under argon. The solvent was removed and the remaining substance chromatographed on silica gel using CH_2Cl_2−hexane (2:1) as eluent. Apart from recovered diphenylsilane, elution furnished a yellow band containing complex **3** (52 mg, 22%): yellow crystals, mp 190 °C dec; ^{1}H NMR (400 MHz, toluene-d_8) δ 2.94 (bs, 2H), 3.76 (bs, 2H), 4.52 (bs, 2H), 4.62 (bs, 2H), 7.15−7.25 (m, 6H), 7.50 (bs, 2H), 7.83 (bs, 2H) ppm; IR (KBr) $\tilde{\nu}_{CO}$ 1969 (s), 1933 (s), 1894 (s), 1881 (s) cm^{-1}; UV (CH_3CN) λ_{max} 283 (ϵ 18 300), 314 (5200); EI-MS (70 eV) *m*/*z* (relative intensity) 794 (M^+, 40), 766 ($M^+ - CO$, 6), 738 ($M^+ - 2CO$, 10), 594 (46), 439 (100). HRMS: calcd for $C_{26}H_{18}SiO_4Re^{187}Re$, 794.006 180; found, 794.006 881. Anal. Calcd for $C_{26}H_{18}SiO_4Re_2$: C, 39.20; H, 2.28. Found: C, 38.96; H, 2.40.

Crystallographic Structure Determination. A summary of crystallographic data is given in Table 1. A suitable crystal was selected and mounted on a glass fiber using Paratone N hydrocarbon oil. Intensity data were collected on a Siemens SMART[28] diffractometer. The unit cell parameters were obtained from a least-squares refinement of 6334 reflections with $I > 10\sigma$ in the range $3.00° < 2\theta < 52.20°$. The frame data were integrated using the program SAINT.[29] The intensities were corrected for Lorentz and polarization effects, and an empirical absorption correction ($T_{max} = 0.623$, $T_{min} = 0.259$) was applied using XPREP.[30] The structure was solved by direct methods (SIR92)[31] and refined by full-matrix least-squares calculations against F^2. Non-hydrogen atoms were treated anisotropically. Hydrogens were included in the model at geometrically calculated positions but not refined. The standard deviation of an observation of unit weight was 0.87, and the maximum shift-to-error ratio was 0.00. The maximum and minimum residual electron densities on the final difference Fourier map corresponded to 1.49 and −1.42 e/Å^3, respectively. Neutral atom scattering factors were taken from Cromer and Waber,[32] anomalous dispersion effects from Creagh and McAuley,[33] and mass attenuation coefficients from Creagh and Hubbel.[34] All calculations were performed using the TeXsan[35] crystallographic software package of Molecular Structure Corp.

Acknowledgment. This paper is dedicated to Professor J. Barluenga (University of Oviedo) on the occasion of his 60th birthday. B.K. is grateful for a postdoctoral fellowship from the Deutsche Forschungsgemeinschaft and M.J.E. for a predoctoral fellowship from the National Science Foundation. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, of the U.S. Department of Energy, under Contract DE-AC03-76SF00098. We thank Professors R. G. Bergman and T. D. Tilley for enlightening words.

Supporting Information Available: Tables of all geometrical and displacement parameters and further details of the X-ray analysis of **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

OM000022U

---

(28) SMART Area Detection Software Package; Siemens Industrial Automation Inc.: Madison, WI, 1995.

(29) SAX Area Detector Integration Program, version 4.024; Siemens Industrial Automation, Inc., Madison, WI, 1995.

(30) XPREP, version 5.03: Part of the SHELXTL Crystal Structure Determination Package; Siemens Industrial Automation, Inc., Madison, WI, 1995.

(31) Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Polidori, G. *J. Appl. Crystallogr.* **1993**, *26*, 343.

(32) Cromer, D. T.; Waber, J. T. In *International Tables for X-ray Crystallography;* Kynoch Press: Birmingham, U.K., 1974; Vol. 4, Table 2.2A.

(33) Creagh, D. C.; McAuley, W. J. In *International Tables for Crystallography*; Wilson, A. J. C., Ed.; Kluwer Academic: Boston, 1992; Vol. C, Table 4.2.6.8, pp 219−222.

(34) Creagh, D. C.; Hubbel, J. H. In *International Tables for Crystallography*; Wilson, A. J. C., Ed.; Kluwer Academic: Boston, 1992; Vol. C., Table 4.2.4.3, pp 200−206.

(35) TeXsan Crystal Structure Analysis Package; Molecular Structure Corp., The Woodlands, TX, 1992.